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TITLE: Oil water type silicone emulsion manufacturing method - involves adding remaining water and diluting after mixing water more than phase inversion point to mixture of polyorganosiloxane and surface active agent

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ABSTRACTED-PUB-NO: JP 10306013A

BASIC-ABSTRACT:

The method involves forming an oil water type silicone emulsion of polyorganosiloxane. Water is added and mixed to a mixture of polyorganosiloxane and a surface active agent by stirring. Remaining water is added and diluted after adding and mixing water of quantity more than a phase inversion point at a time, in the mixture of polyorganosiloxane and surface active agent.

USE - In manufacture O/W type siloxane emulsion used for defoaming, release, slip, feel improvement in various fields including cosmetics and fragrance product fields such as hair cosmetics

ADVANTAGE - Obtains silicone emulsion with excellent emulsion stability and proportioning stability by promoting close particle distribution. Obtains emulsion of large particle diameter which was difficult to obtain by other processes. Facilitates simple and easy control of particle diameter. Eliminates influence by variety and scale of equipment. Shortens manufacturing process.

---------- KWIC ---------

Title - TIX (1):

Oil water type silicone emulsion manufacturing method - involves adding remaining water and diluting after mixing water more than phase inversion point to mixture of polyorganosiloxane and surface active agent

Standard Title Terms - TTX (1):

OIL WATER TYPE SILICONE EMULSION MANUFACTURE METHOD ADD REMAINING WATER DILUTE AFTER MIX WATER MORE PHASE INVERT POINT MIXTURE SURFACE ACTIVE AGENT

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(54) SILICONE EMULSION, ITS PRODUCTION AND HAIR COSMETIC

(57)Abstract:
PROBLEM TO BE SOLVED: To obtain the subject O/W-type emulsion having easily controllable particle diameter, a narrow particle diameter distribution and excellent emulsion stability and compounding stability in spite of large particle diameter by adding water in an amount larger than the phase-conversion volume at a stroke to a mixture of a polyorganosiloxane and a surfactant, stirring the product and diluting with the remaining part of water.
SOLUTION: An emulsion having a relatively large particle diameter (e.g. 1-100 μm) difficult to get a high stability can be produced by mixing a mixture of (A) a polyorganosiloxane [e.g. a compound of the formula RaSiO(4-a)/2 (R is a univalent hydrocarbon group; (a) is a positive number smaller than 4), etc.] and (B) a surfactant (e.g. a polyoxyethylene fatty acid ester) with 1-5 times volume of water based on the volume of water at a phase-conversion point and diluting the product by adding the remaining part of water. The amount of the component B is preferably 1-30 pts.wt. based on 100 pts.wt. of the component A.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] Glazing of this invention is carried out and it relates to the O/W mold silicone emulsion used in various fields for the purpose of defoaming, mold release, slipping, feel amelioration, etc., and its manufacture approach. It is related with the silicone emulsion especially used suitably by cosmetics / perfumery-and-cosmetics field for the purpose, such as improvement in a feel, and its manufacture approach.
[0002]
[Description of the Prior Art] From the former, many silicone emulsions are used in order to give feels, such as smoothness and gloss, to hair and the skin. Since what adheres to hair or the skin promptly, and does not flow and fall at the time of use was advantageous when making the description of silicone give, it was expected that the property which was excellent, so that emulsion particle diameter was large was acquired by the property top, but when particle diameter was enlarged, and the stability of an emulsion fell and it blended with various products further, there was a problem that combination stability with them fell remarkably. An emulsion has the inclination for the stability to worsen so that particle diameter is generally large, but when this tends to enlarge particle diameter, the particle size distribution becomes large, turns into particle size distribution with further two or more peaks in many cases, and is considered to have caused the stability fall with this going too far. therefore, current -- the particle diameter of a silicone emulsion -- less than 1 micrometer -- many things of small particle diameter are used comparatively. Some approaches are learned in order to control emulsion particle diameter until now. For example, there is the approach of choosing the class and HLB of a surfactant. In this case, in order to obtain the emulsion of the target particle diameter according to the silicone to be used, even if it sees from the numerousness of the classes of surfactant by which current marketing is carried out, there is a problem that there are very many mark which should be examined and examination takes a long period of time to them so that clearly. Moreover, conversely, in cosmetics / perfumery-and-cosmetics field, the surfactant which can be used is restricted and there was a problem that it was difficult to obtain the emulsion of the property it can fully be satisfied only with those combination of a property. As the concrete technique of controlling the particle diameter of an emulsion The HLB method (1:311 Griffin, wc:J.Soc.Cosmet.Chem., 1949), liquid crystal emulsification method (Washitani et al.: oil chemistry, 30, 38, 1981) Phase inversion emulsification method (T. J.Lin, J.Soc.Cosmet.Chem., 30 167,976) And D-phase emulsification method (Washitani et al.: the Chemical Society of Japan, 1399, 1983) etc. -- various things are proposed. However, these emulsification methods are for mainly obtaining a less than 1-micrometer small particle diameter article, and cannot say it as the effective means for controlling the particle diameter of a large particle diameter article like this invention. Of course, although a thing 1 micrometers or more may be obtained depending on the surfactant to be used, there were troubles, such as above-mentioned stability. How to change the share which starts at the time of emulsification by changing the agitating speed at the time of the emulsification which changes the class of emulsifier which changes the temperature at the time of emulsification as other approaches of furthermore

controlling particle diameter etc. can be considered. However, it will become a thing with particle size distribution with two or more peaks [**** / that distribution of the particle diameter of the emulsion done since a difference arises / the share which control exact / of temperature or agitating speed / by this approach is difficult, and also starts at the time of emulsification / by no means uniformly in / whole / an emulsification system partially in the impeller circumference etc. becomes large]. If it becomes like this, the stability of an emulsion and combination stability with other ingredients will fall remarkably. This inclination becomes remarkable [a large particle diameter article] especially. Although the particle diameter of an emulsion becomes large so that the share which this requires at the time of stirring is small, it is because it is difficult to apply a small share to homogeneity at the whole. Moreover, in it becoming difficult to give a uniform share's into [whole] an emulsification system on the occasion of actual manufacture so that a manufacture scale is large, when the manufacturing installation changed or the scale changed, even if conditions, such as temperature and a rotational frequency, were the same, it became different particle diameter in many cases, and there was a trouble that it will be necessary to examine manufacture conditions, such as optimal temperature and a rotational frequency, each time.

[0003]

[Objects of the Invention] without the purpose of this invention solves said trouble and it changes an emulsion presentation -- the particle diameter -- easy -- controlling -- in addition -- and while the particle size distribution acquired is narrow and has large particle diameter, it is in obtaining the emulsion excellent in emulsion stability and combination stability.

[0004]

[Elements of the Invention] this invention persons came to complete a header and this invention for the particle diameter obtained by that the narrow emulsion of particle size distribution is obtained and changing further the amount of the water added first being controllable by adding the water more than a phase-inversion point to the mixture of silicone and a surfactant at a time, and diluting with the remaining water after mixed stirring, as a result of repeating examination wholeheartedly, in order to attain the above-mentioned purpose. That is, for this invention, the emulsion mean particle diameter which in manufacturing the O/W mold silicone emulsion of polyorganosiloxane added the water more than a phase-inversion point at a time into the mixture of polyorganosiloxane and a surfactant, and be obtained after mixed stirring by the manufacture approach of the silicone emulsion characterize by adding and diluting the remaining water and this manufacture approach be 1-100. It be relate with the silicone emulsion which be mum, and the hair cosmetics which come to contain this silicone emulsion in a list.

[0005]

[Embodiment of the Invention] Hereafter, this invention is explained to a detail. The polyorganosiloxane used for this invention can use a well-known thing conventionally. For example, what is shown by general formula RaSiO (4-a) / 2 (a permutation or an unsubstituted monovalent hydrocarbon radical, and a show less than four positive number here in R) is illustrated. Inside R of a formula Even if it is a permutation or an unsubstituted monovalent hydrocarbon radical and is the same You may be the thing of two or more classes. For example, a methyl group, an ethyl group, a hexyl group, Aralkyl radicals, such as aryl groups, such as alkyl groups, such as a decyl group, and a phenyl group, and 2-methylphenylethyl radical, Amino content radicals, such as 3-aminopropyl radical and a 2-aminoethyl-3-aminopropyl radical, Fluorine content radicals, such as polyoxyalkylene groups, such as a polyoxyethylene radical, a polyoxypropylene radical, and a polyoxyethylene-polyoxypropylene radical, 3 and 3, and 3-trifluoro propyl group, etc. are illustrated. These are various R to the appearance that a methyl group is desirable when the purpose used and the thing which suited the application can be chosen, for example, it aims at the dry feel grant to hair in hair cosmetics, an amino-group content radical is desirable for the admiration grant effectiveness or the purpose of the improvement in adhesion gently, and an aryl group is desirable for the purpose of the improvement in gloss. It can choose. Moreover, a It is less than four positive number, and is the ease of carrying out of emulsification, the usefulness of the siloxane obtained, etc. to 1.0-2.5. It is desirable. The number of the polyorganosiloxanes to be used may be one, or that of them may be [two or more]. for example, the

viscosity which has a cuticle protective effect in addition to giving the feel excellent in hair -- 1 million -- cSt(s) Generally it is performed that it uses the poly dimethylsiloxane of hundreds cP(s) together in a the very thing simple substance in order that the poly dimethylsiloxane to exceed may have the bad breadth to hair and it may compensate it with this. Moreover, hundreds cSt(s) For example, the isoparaffin system solvent may be used instead of poly dimethylsiloxane. Of course, the manufacture approach of this invention is applicable also in this case. Viscosity since particle size distribution is small, an emulsion with good stability is obtained efficiently, and grant of the good feel to hair and grant of a good cuticle protective effect are performed in the emulsion constituent obtained in the manufacture approach of this invention when it is used for hair cosmetics is 50,000-50,000,000cSt especially 10,000 to 100,000,000 cSt. It is desirable to use poly dimethylsiloxane.

[0006] As a surface active agent used for this invention, any of the Nonion system surface active agent, an anion system surface active agent, and a cation system surface active agent are sufficient. As an Nonion system surface active agent, mono-lauric-acid glyceryl, mono-myristic-acid glyceryl, A glycerine fatty acid ester like monostearin acid glyceryl and mono-oleic acid glyceryl; The polyglyceryl fatty acid ester which has the same fatty-acid residue, A sorbitan fatty acid ester, polyoxyethylene glycerine fatty acid ester, Polyoxyethylene sorbitan fatty acid ester and polyoxyethylene fatty acid ester; The polyoxyethylene lauryl ether, The polyoxyethylene cetyl ether, polyoxyethylene stearylether, Polyoxyethylene alkyl ether like the polyoxyethylene oleyl ether; Polyoxyethylene octyl phenyl ether, Polyoxyethylene alkyl phenyl ether like the polyoxyethylene nonylphenyl ether; oxyethylene oxypropylene copolymer; and polyether denaturation poly dimethylsiloxane are illustrated. As an anion system surface active agent, hexyl benzenesulfonic acid sodium, Octyl benzenesulfonic acid sodium, DESHIRU benzenesulfonic acid sodium, Sodium dodecylbenzenesulfonate, cetyl benzenesulfonic acid sodium, Alkyl naphthyl sulfonic-acid sodium like alkyl-benzene-sodium-sulfonate; butyl naphthyl sulfonic-acid sodium like Millis Chill benzenesulfonic acid sodium; Polyoxyethylene octyl ethereal sulfate ester sodium, Polyoxyethylene DESHIRU ethereal sulfate ester sodium, Polyoxyethylene-alkyl-ether sulfate sodium salt like polyoxyethylene eicosyl ethereal sulfate ester sodium; Polyoxyethylene monochrome like polyoxyethylene DESHIRU phenyl ether sulfate sodium (Alkylphenyl) Ethereal sulfate ester sodium salt etc. is illustrated. As a cation system surface active agent, quarternary ammonium salt like octyl trimethylammonium chloride, dodecyl trimethylammonium chloride, hexadecyl trimethylammonium chloride, beef tallow trimethylammonium chloride, palm oil trimethylammonium chloride, octyl dimethylbenzyl ammonium chloride, DESHIRU dimethylbenzyl ammonium chloride, and dioctadecyl dimethyl ammonium chloride is illustrated. These surfactants can usually use together two or more sorts of things, the same network or a different network. In the manufacture approach of this invention, particle size distribution is narrow, it is desirable to use what contains one or more kinds of Nonion system surfactants at least from grant of the good feel to hair being performed, when an emulsion with good stability is obtained and uses it for hair cosmetics in the emulsion constituent obtained, and it is desirable to use only especially the Nonion system surfactant. As an Nonion system surface active agent, although mentioned above, polyoxyethylene sorbitan fatty acid ester, polyoxyethylene fatty acid ester, and polyoxyethylene alkyl ether are used preferably especially. A surfactant is polyorganosiloxane. 1 - 30 weight section is used to the 100 weight sections. This is because emulsification is fully impossible in case of under 1 weight section, and it may not be enough demonstrated on the other hand since the amount of a surfactant has many properties of silicone original when using the emulsion which will be obtained if 30 weight sections are exceeded. desirable -- polyorganosiloxane the 100 weight sections -- receiving -- 2 - 20 weight section -- 5-15 weight section use is carried out especially preferably.

[0007] Heating an oil phase component and a surfactant if needed, mixing, and usually adding water here gradually conventionally, when preparing an O/W mold emulsion, it mixes further and is obtained. In this case, emulsifiers, such as a colloid mill, a homomixer, and a pressurization homogenizer, are used if needed. For example, in the phase inversion emulsification method mentioned above as a conventional technique, water is gradually added to the mixture of an oil phase component and a surfactant. First, D phase is made to form after going via a W/O mold. D phase is also called a surfactant

phase, an oil and water are the liquid crystal phases arranged in the sandwiches mold with the lamellae phase of a surface active agent; and, as for an exterior, transparency comes out here. Usually, this point is made into the phase-inversion point. An oil droplet will be in the condition of having distributed finely, adds water to infinity further here, and makes an O/D emulsion form in it in this liquid crystal phase. At this time, a continuous phase turns into D phase, without influencing the oil phase which is a dispersed phase. Furthermore water is added and an O/W mold emulsion is made. By carrying out like this, the O/W mold emulsion which the oil droplet distributed minutely is obtained. By this approach, water is added gradually, it is going to obtain the O/W mold emulsion of uniform small particle diameter by making the inside of an emulsification system uniform for every phase, and the count of irrigation usually becomes 5 - 10 times in fact 3 times or more. In this case, in order to make the inside of a system into homogeneity, sufficient stirring is needed and manufacture takes long duration each time. Moreover, D phase is a usually hard gel object, stirring effectiveness high for considering as homogeneity and big power are needed, and it becomes difficult to make the inside of a system into homogeneity, so that hyperviscosity [an emulsified object]. In this case, a place inadequate in stirring tends to exist partially, and particle diameter varies as a result. Since there is such a thing, the particle size distribution of the emulsion which will be obtained if equipment and a scale change tends to change, and there is a problem that control is difficult. In addition, beforehand, the amount of the water used as a phase-inversion point can repeat small quantity, in addition stirring for water in the condition of having maintained at constant temperature, into the mixture of an oil phase component and a surfactant, and can measure them by the approach of evaluating the point that the transparency of contents is the highest, as a phase-inversion point.

[0008] Although characterized by adding the water more than a phase-inversion point at a time at the mixed stock of an oil phase component and a surfactant in this invention, this amount of irrigation is more than an amount used as the aforementioned D phase. Since the amount of the water with which D phase is formed changes with the oil phase component to be used or surfactants, limitation of specifying the amount of irrigation to an oil phase component, for example is difficult. In this invention, first time irrigation of the water of the amount of one to 5 times of the amount is carried out on the basis of the amount of water of a phase-inversion point. Even if this adds a dilution water as it is less than 1 time, in order that mixture of an oil phase component, a surfactant, and water cannot distribute to homogeneity or may distribute, it is for requiring long duration. Moreover, if water is poured into the first time, in order that mixture of an oil phase component and a surfactant cannot distribute in water or may distribute too the water more than the amount of 5 times, it is for requiring long duration. in order to distribute easily at both an irrigation process and a dilution process and to acquire uniform particle size distribution -- desirable -- the amount of 1.1 to 3 times -- especially -- desirable -- It is the amount of water of the amount of 1.5 - 2 double. If a dilution water is added after the particle diameter which is obtained according to this invention pours water in the amount of water of a phase-inversion point, it will become a small thing, and the particle diameter obtained becomes large gradually as the amount will become large, if water is poured at a time in the water more than a phase-inversion point. That is, it becomes possible to control the particle diameter of the emulsion obtained by changing the amount of first time irrigation. In this invention, fundamentally, since water is only poured in 2 steps in water, a production process can be shortened. Moreover, in order not to make a hard gel phase form, it is easy to mix the inside of a system to homogeneity. That is, it is hard to be influenced of equipment or a scale. This approach has few production processes, and since it is hard to be influenced of equipment or a scale, batch type manufacture is the approach for which it was suitable also when continuation manufacture was carried out, of course.

[0009] In the manufacture approach of the emulsion of this invention As mentioned above, as an ingredient which may emulsify by using together, may use an isoparaffin system solvent together in this way, and may blend it, polyorganosiloxane The emulsion obtained for for example, the purpose, such as diluting polyorganosiloxane, getting wet and giving breadth nature, when it is used for hair cosmetics Various solvents, such as chlorine-based solvents, such as isoparaffin system solvents, such as aromatic solvents, such as paraffin series solvents, such as a hexane and a heptane, toluene, and a xylene, an iso

heptane, and an isooctane, and chloroform, an ester solvent, and a glycol system solvent, are illustrated. moreover, the defoaming purpose of the emulsion obtained -- silica powder -- moreover, as for waxes, such as carnauba wax, thickeners, such as antiseptics, such as propyl para hydroxybenzoate, a sodium benzoate, a citric acid, and a salicylic acid, an alginic acid, gum arabic, casein, methyl cellulose, hydroxyethyl cellulose, a cation denaturation etherification cellulose, hydroxypropyl DIN powder, polyvinyl alcohol, a polyvinyl pyrrolidone, and a bentonite, etc. can use solid-state powder, such as a zeolite, together further for the purpose of the improvement in gloss for the purpose of thixotropic grant. Also when these are used together with polyorganosiloxane, the manufacture approach of this invention can be applied.

[0010] The emulsion obtained by the manufacture approach of this invention has the description that it is narrow at mono dispersion [particle size distribution], and has the description of excelling in the stability of an emulsion. Therefore, when it is used as ingredients, such as cosmetics, there is usefulness of excelling in the shelf life of the cosmetics obtained. Generally by the manufacture approach of this invention, mean particle diameter 0.1-200 Obtaining what has stability good until now, although the emulsion of mum is obtained efficiently is the difficult thing, 1-100, which has comparatively large particle diameter. There are mum and the description that a 3-80-micrometer thing is obtained especially efficiently. [for example,] When it blends with cosmetics etc., the emulsion with such large particle diameter obtained by the manufacture approach of this invention is not only excellent in the stability, but has the description that the property which was excellent in silicone original is demonstrated.

[0011]

[Effect of the Invention] According to the manufacture approach of this invention, there is the description that a silicone emulsion with good stability with narrow particle size distribution is obtained, and a silicone emulsion with good stability with the large particle diameter which was difficult to manufacture until now is obtained especially. It is easy to control the particle diameter of an emulsion furthermore, and it cannot be easily influenced by the class or scale of equipment, and there is the description that it can carry out by the production process shortened rather than the conventional manufacture approach.

[0012]

[Example] An example is given to below and this invention is explained in more detail. In addition, "% of the weight" is expressed"%" in an example. Moreover, viscosity shows the value in 25 degrees C. The presentation of the emulsion used in the example is shown in Table 1. Moreover, the particle diameter of an emulsion was measured by the approach shown below. Moreover, in each emulsion presentation, it measured by the approach of showing a 70-degree C phase-inversion point below. the phase-inversion point obtained here -- amount of water is shown in Table 1.

[0013]

[Table 1]

成 分	組成－1	組成－2	組成－3	組成－4
ポリジメチルシロキサン1	36%	36%	44%	—
ポリジメチルシロキサン2	—	—	—	60%
ポリジメチルシロキサン3	24%	24%	30%	—
アミノ基含有シロキサン	—	6%	—	—
ノニオン系界面活性剤1	1.2%	1.8%	—	1.5%
ノニオン系界面活性剤2	—	1.1%	1.5%	—
ノニオン系界面活性剤3	3.8%	3.1%	—	2.0%
ノニオン系界面活性剤4	—	—	—	2.5%
カチオン系界面活性剤1	—	—	0.5%	—
水	35%	28%	24%	34%
転相点水量	7%	7%	5%	9%

[0014]
Notes
poly dimethylsiloxane 1:200cSt poly dimethylsiloxane Poly dimethylsiloxane 2:10** cSt Poly dimethylsiloxane Poly dimethylsiloxane 3:2000** cSt Poly dimethylsiloxane Amino-group content siloxane :1000cSt, nitrogen atom content 0.9% of 2-AMINOE Chill-3-aminopropyl radical content poly JIMECHIRUSHI Loki Sun The Nonion system surface active agent 1 : The polyoxyethylene (23) lauryl ether The Nonion system surface active agent 2 : The polyoxyethylene (6) lauryl ether The Nonion system surface active agent 3 : Polyoxyethylene (55) monostearate Nonion system surface active agent 4 : Polyoxyethylene (20) sorbitan mono-steer rate Cation system surface active agent 1 : Cetyl trimethylammonium chloride emulsion particle diameter measurement coal tar company make and LS mold (measurement principle: laser diffraction / dispersion technology) were used. From the obtained result, mean particle diameter, most frequent particle diameter, and mean particle diameter / most frequent particle diameter were checked. In addition, the breadth of particle size distribution was judged by the ratio of mean particle diameter / most frequent particle diameter. That is, distribution judged that it was narrow at mono dispersion, so that this ratio was close to 1.
- a phase-inversion point -- after stirring under heating and water were added for the measurement siloxane component and activator component of amount of water the amount every 1% at 70 degrees C, and stirring was repeated. this time -- the point that the transparency of contents is the highest -- a visual judgment -- carrying out -- the amount of irrigation at that time -- a phase-inversion point -- it considered as amount of water.
[0015] The example 1 emulsion presentation -1 is totaled. Each component was used so that it might be set to 100g. 200 cc Into the flask, the mixture (36g of poly dimethylsiloxane 1 and 24g) of poly dimethylsiloxane 3 was put in, and it heated at 70 degrees C. Also after this, 70-degree C heating was continued until it added the water of the whole quantity. It is the Nonion system surfactant 1 here. 1.2g and 3.8g of the Nonion system surfactant 3 were added, and manual stirring was carried out using the metal spatula. When stirring was continued for about 5 minutes, with 70-degree C heating maintained, the Nonion system surface active agent fused and it was mixed with poly dimethylsiloxane. 8g of water was added here as first time irrigation, and stirring was continued further. Stirring was continued for about 10 minutes and the transparent gel object was obtained. Next, the 27g of the remaining water was added as a dilution water, stopping and cooling 70-degree C heating radiationally, it stirred for about 10 minutes and the emulsion of opalescence was obtained. The mean particle diameter of this emulsion, most frequent particle diameter, and mean particle diameter / most frequent particle diameter are shown in Table 2.

It was operated like the example 1 except having set the amount of example 2 first-time irrigation to 10g, and having set the amount of dilution waters to 25g. The opalescence liquid which has transparency a little [viscous] after first time irrigation and stirring was obtained, and, finally the emulsion of opalescence was obtained. Evaluation results, such as particle diameter, are shown in Table 2.
It was operated like the example 1 except having set the amount of example 3 first-time irrigation to 12g, and having set the amount of dilution waters to 23g. The opalescence liquid which has transparency a little [viscous] after first time irrigation and stirring was obtained, and, finally the emulsion of opalescence was obtained. Evaluation results, such as particle diameter, are shown in Table 2.
It was operated like the example 1 except having set the amount of example 4 first-time irrigation to 15g, and having set the amount of dilution waters to 20g. After first time irrigation and stirring, the viscous opalescence liquid was obtained and, finally the emulsion of opalescence was obtained. Evaluation results, such as particle diameter, are shown in Table 2.
It was operated like the example 1 except having set the amount of example 5 first-time irrigation to 18g, and having set the amount of dilution waters to 17g. After first time irrigation and stirring, the viscous opalescence liquid was obtained and,.finally the emulsion of opalescence was obtained. Evaluation results, such as particle diameter, are shown in Table 2.
It was operated like the example 1 except having set the amount of example 6 first-time irrigation to 22g, and having set the amount of dilution waters to 13g. After first time irrigation and stirring, the viscous opalescence liquid was obtained and, finally the emulsion of opalescence was obtained. Evaluation results, such as particle diameter, are shown in Table 2.
Each component was used so that the example 7 emulsion presentation -1 might be set to a total of 1500g. To the horse mackerel homomixer (Product made from the formation of a special opportunity) equipped with the 2l. container, it is poly dimethylsiloxane 1. 540g and poly dimethylsiloxane 3 360g mixture was put in and it heated at 70 degrees C. This heating was continued to dilution-water addition. Moreover, rotational frequency of a paddle type mixer It was referred to as 60rpm and stirring was continued. This stirring condition was continued to the last emulsion draw. 18g of the Nonion system surfactant 1 and 48g of the Nonion system surfactant 3 were added here, stirring was continued for about 20 minutes, the Nonion system surfactant was fused, and it mixed with poly dimethylsiloxane. It is water as first time irrigation here. In addition, 225g of stirring was continued further. Stirring was continued for about 20 minutes and the transparent gel object was obtained. Next, the remaining water as a dilution water 300g was added, it stirred for about 20 minutes, the emulsion of opalescence was prepared, and it was similarly estimated as the example 1. In addition, this example 7 becomes that which the example 4 scaled up [which scaled up and equipment-changed].
Rotational frequency of example 8 paddle type mixer Except having made it 15rpm, it was operated like the example 7, the emulsion of opalescence was prepared, and it evaluated similarly.
[0016] The example of comparison 1 emulsion presentation -1 is totaled. Each component was used so that it might be set to 100g. Into 200 cc flask, the mixture (36g of poly dimethylsiloxane 1 and 24g) of poly dimethylsiloxane 3 was put in, and it heated at 70 degrees C. Also after this, 70-degree C heating was continued until it added the water of the whole quantity. 1.2g of the Nonion system surfactant 1 and 3.8g of the Nonion system surfactant 3 were added here, and manual stirring was carried out using the metal spatula. Stirring was continued for about 5 minutes, the Nonion system surface active agent was fused, with 70-degree C heating held, and it mixed with poly dimethylsiloxane. 2g of water was added here as first time irrigation, and stirring was continued for about 10 minutes. The actuation which furthermore adds 2g of water and is stirred for 10 minutes was repeated twice. Here, what had the shape of opalescence oil viscous until now became a transparent gel object. The actuation which furthermore adds 2g of water and is stirred for 10 minutes was repeated 8 times, then, 13g of water was added as a dilution water, stirring was continued, the emulsion of opalescence was prepared, stopping and cooling 70-degree C heating radiationally, and it was similarly estimated as the example 1.
After the stirring termination after carrying out example of comparison 2 first-time irrigation, except having stopped 70-degree C heating, it was operated like the example 1 of a comparison, the emulsion was prepared, and it evaluated similarly.

Each component was used so that the example of comparison 3 emulsion presentation -1 might be set to a total of 1500g. To the horse mackerel homomixer (Product made from the formation of a special opportunity) equipped with the 2l. container, it is poly dimethylsiloxane 1. 540g and poly dimethylsiloxane 3 360g mixture was put in and it heated at 70 degrees C. This heating was continued to dilution-water addition. Moreover, rotational frequency of a paddle type mixer It was referred to as 60rpm and stirring was continued. This stirring condition was continued to the last emulsion draw. 18g of the Nonion system surfactant 1 and 48g of the Nonion system surfactant 3 were added here, stirring was continued for about 20 minutes, melting of the Nonion system surfactant was carried out, and it mixed with poly dimethylsiloxane. 30g of water was added here as first time irrigation, and stirring was continued for about 20 minutes. The actuation which furthermore adds 30g of water and is stirred for 20 minutes was repeated twice. Here, what had the shape of opalescence oil viscous until now became a transparent gel object. The actuation which adds 30g of water here and is stirred for 20 minutes is repeated 8 times, and then it is water as a dilution water. 195g was added, stirring was continued, the emulsion of opalescence was prepared, stopping and cooling 70-degree C heating radiationally, and it was similarly estimated as the example 1. In addition, this example 3 of a comparison becomes that which the example 1 of a comparison scaled up [which scaled up and equipment-changed].
Rotational frequency of example of comparison 4 paddle type mixer Except having been referred to as 15rpm, it was operated like the example 3 of a comparison, the emulsion was prepared, and it evaluated similarly.
It was operated like the example 1 except having set the amount of example of comparison 5 first-time irrigation to 4g, and having set the amount of dilution waters to 31g. Although the opalescence liquid which has transparency a little [viscous] after first time irrigation and stirring was obtained, in stirring after dilution-water addition, a dilution water did not get used and the emulsion of opalescence was not able to be obtained.
[0017] The example 9 emulsion presentation -2 is totaled. Each component was used so that it might be set to 100g. Into 200 cc flask, the mixture (36g of poly dimethylsiloxane 1, 24g of poly dimethylsiloxane 3, and 6g) of an amino-group content siloxane was put in, and it heated at 70 degrees C. It is the Nonion system surfactant 1 here. 1.8g and the Nonion system surfactant 2 1.1g and the Nonion system surfactant 3 3.1g was added and manual stirring was carried out using the metal spatula. Stirring was continued for about 5 minutes, melting of the Nonion system surface active agent was carried out, and it mixed with poly dimethylsiloxane. 8g of water was added here as first time irrigation, and stirring was continued further. When stirring was continued for about 10 minutes, it became a transparent gel object. Next, the 20g of the remaining water was added as a dilution water, it stirred for about 10 minutes, the emulsion of opalescence was prepared, and it was similarly estimated as the example 1.
It was operated like the example 9 except having set the amount of example 10 first-time irrigation to 20g, and having set the amount of dilution waters to 8g. After first time irrigation and stirring, the viscous opalescence liquid was obtained and, finally the emulsion of opalescence was obtained. Evaluation results, such as particle diameter, are shown in Table 2.
The example 11 emulsion presentation -3 is totaled. Each component was used so that it might be set to 100g. Into 200 cc flask, the mixture (44g of poly dimethylsiloxane 1 and 30g) of poly dimethylsiloxane 3 is put in, and it is the Nonion system surface active agent 2 here. 1.5g and cation system surface active agent 2 0.5g was added and manual stirring was carried out using the metal spatula. It mixed by continuing stirring for about 5 minutes. 6g of water was added here as first time irrigation, and stirring was continued further. When stirring was continued for about 10 minutes, it became a transparent gel object. Next, the 18g of the remaining water was added as a dilution water, it stirred for about 10 minutes, the emulsion of opalescence was prepared, and it was similarly estimated as the example 1.
It was operated like the example 11 except having set the amount of example 12 first-time irrigation to 15g, and having set the amount of dilution waters to 9g. After first time irrigation and stirring, the viscous opalescence liquid was obtained and, finally the emulsion of opalescence was obtained. Evaluation results, such as particle diameter, are shown in Table 2.

The example 13 emulsion presentation -4 is totaled. Each component was used so that it might be set to 100g. 60g of poly dimethylsiloxane 2 was put in into 200 cc flask, and it heated at 70 degrees C. It is the Nonion system surfactant 1 here. 1.5g, 2g of the Nonion system surfactant 3, and the Nonion system surfactant 4 2.5g was added and manual stirring was carried out using the metal spatula. It mixed by continuing stirring for about 5 minutes. 10g of water was added here as first time irrigation, and stirring was continued further. When stirring was continued for about 10 minutes, it became a transparent gel object. Next, the 24g of the remaining water was added as a dilution water, it stirred for about 10 minutes, the emulsion of opalescence was prepared, and it was similarly estimated as the example 1.
It was operated like the example 13 except having set the amount of example 14 first-time irrigation to 15g, and having set the amount of dilution waters to 19g. After first time irrigation and stirring, the viscous opalescence liquid was obtained and, finally the emulsion of opalescence was obtained. Evaluation results, such as particle diameter, are shown in Table 2.
[0018]
[Table 2]

No	平均粒子径 (μm)	最頻粒子径 (μm)	平均粒子径／最頻粒子径
実施例1	1.64	1.59	1.03
実施例2	2.56	2.54	1.01
実施例3	3.25	3.14	1.04
実施例4	3.75	3.69	1.02
実施例5	5.99	6.45	0.92
実施例6	6.43	7.08	0.91
実施例7	3.65	3.45	1.06
実施例8	3.82	3.70	1.03
比較例1	5.51	4.71	1.17
比較例2	1.60	1.51	1.06
比較例3	3.64	3.57	1.02
比較例4	7.81	15.1	0.52
実施例9	1.78	1.81	0.98
実施例10	7.34	7.59	0.97
実施例11	24.5	22.5	1.09
実施例12	46.4	42.8	1.08
実施例13	11.4	10.3	1.11
実施例14	56.8	54.9	1.03

[0019] Although examples 1-6 changed the first-time amount of irrigation, the particle diameter obtained changed and the distribution was small. Moreover, although an example 7 is the same combination as an example 4 and equipment and a scale were changed, the particle size distribution acquired was the same. Furthermore, although the example 8 changed the rotational frequency of stirring with an example 7, this equipment, and a scale, it was the same. [of the particle size distribution acquired too] On the other hand, although the particle diameter obtained at emulsification temperature changed in the examples 1 and 2 of a comparison, in the high example 1 of a comparison of temperature, distribution became large. Moreover, although the examples 3 of a comparison are the same combination as the example 1 of a comparison, and the same temperature and equipment and a scale

were changed, the obtained particle diameter became small. Furthermore, the particle diameter of the example 4 of a comparison obtained although the rotational frequency of stirring with the example 3 of a comparison, this equipment, and a scale was changed was large, and the distribution became extremely large. Moreover, in the examples 9-14, the system from which a surfactant and polyorganosiloxane differ could also change particle diameter by changing the amount of first time irrigation, and the particle size distribution was comparatively narrow.
[0020] The example at the time of using the emulsion of this invention for cosmetics is shown below.
Example 15 (hair set constituent)
The emulsion prepared in the example 1 was used, and the hair set constituent of the following presentations was prepared and evaluated. A result is shown in Table 3.
- Presentation silicone emulsion (example 1) 5.0 % hydroxyethyl cellulose 0.2 % ethyl alcohol 10.0 % perfume 0.2 % ion exchange water After applying 4g of hair set constituents to 2g of hair with a die length [84.6 % and evaluation approach <ductility> die length] of 25cm from the distance of 20cm at homogeneity, the lie of hair was arranged with the commercial comb before desiccation, and it dried, having applied this hair for 60 minutes by 40-degree C warm air, and evaluated in accordance with the valuation basis shown below. O -- When it touches by hand, there is nothing with ** and it is a natural feel.
** -- When it touches by hand, it is with [a little] **.
x -- When it touches by hand, it is severe with ** and there is a hard feel.
After applying 4g of hair set constituents to 2g of hair with a <smoothness> die length of 25cm from the distance of 20cm at homogeneity, it dried having applied this hair for 60 minutes by 40-degree C warm air, and evaluated in accordance with the valuation basis which shows the condition as that comb below using a commercial comb. O -- There is no connection and a comb passes smoothly.
** -- Although there is connection, it can let a comb pass.
x -- A comb does not pass.
It is the outer diameter of 1.2cm about this hair after applying 4g of hair set constituents to 2g of hair with a <set holding power> die length of 25cm from the distance of 20cm at homogeneity. It wound around the curler, and by 40-degree C warm air, it applied for 60 minutes and dried. Then, the die length (L1) immediately after removing hair and hanging from a curler, perpendicularly in the ambient atmosphere of the temperature of 30 degrees C and 80% of relative humidity and the die length after 1-hour neglect (L2) were measured, and the curl retention was computed from the degree type. curl retention (%) =(25-L2)/(25-L1) x100 -- in addition, the curl retention made success (front Naka, "O" mark) and less than 60% of thing the rejection (front Naka, "x" mark) for 60% or more of thing.
[0021] The emulsion prepared in the example 16 example 4 was used, and also the hair set constituent was prepared and evaluated like the example 15. A result is shown in Table 3.
The emulsion prepared in the example 17 example 7 was used, and also the hair set constituent was prepared and evaluated like the example 15. A result is shown in Table 3.
The emulsion prepared in the example 18 example 9 was used, and also the hair set constituent was prepared and evaluated like the example 15. A result is shown in Table 3.
The emulsion prepared in the example 19 example 11 was used, and also the hair set constituent was prepared and evaluated like the example 15. A result is shown in Table 3.
The emulsion prepared in the example 1 of example of comparison 6 comparison was used, and also the hair set constituent was prepared and evaluated like the example 15. A result is shown in Table 3.
The emulsion prepared in the example 4 of example of comparison 7 comparison was used, and also the hair set constituent was prepared and evaluated like the example 15. A result is shown in Table 3.
[0022]
[Table 3]

	実施例					比較例	
	15	16	17	18	19	6	7
しなやかさ	○	○	○	○	○	×	○
なめらかさ	○	○	⊙	○	○	△	△
セット保持力	○	○	○	○	○	○	×

[0023] An example 20, the example 8 (shampoo constituent) of a comparison
The emulsion prepared in the example 1 or the example 1 of a comparison was used, and two kinds of shampoo constituents of the following presentations were prepared, and were evaluated. - Presentation Silicone emulsion (an example 1 or example 1 of a comparison) 3.0 % polyoxyethylene lauryl ether 15.0 % lauric-acid diethanolamide 5.0 % perfume 0.2 % ion exchange water The shampoo constituent (example 20) which used the emulsion of 76.8 % examples 1 could give to hair the feel which gave good luster and felt refreshed, and its stability was also good. On the other hand, the shampoo constituent (example 8 of a comparison) which used the emulsion of the example 1 of a comparison had the inadequate grant and stability of the luster to the hair of a thing which can give the feel of good extent a little.

[Translation done.]

pages 8 + 9 actually describe a prior art method mirroring that of the claims but it says that the method is disadvantageous because a condition is reached where enormous shear forces are needed to homogenize the system, homogeneity being indicated by narrow particle size distribution

↓

ostensibly would be even worse for high viscosity polymers

this was done in Ex

seems to be a suggestion [0005] that where silicone of viscosity > 1000000 is used, a low mw silicone or isoparaffin should be added

the invention of 10-30COL3 contemplates adding water in 2 stages like the claims but it is not entirely clear that the amount of water added 1st, 1-2 times the qty needed of the phase inversion point, is consistent w/ that mentioned by the claims

↓

not clear that 8 g represents the minimum for phase inversion

in Ex 1, 8 g of water are added to 60 g silicone which means the amt of water is 8/60 or 13 wt%

~~to~~ Ex are done on a small scale w/ no mention of extruder in broader disclosure

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A SILICONE EMULSION, THE MANUFACTURING METHOD THEREFORE, AND A HAIR COSMETIC

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Claims /2*

1. A method for manufacturing a silicone emulsion, being a method for manufacturing a polyorganosiloxane O/W silicone emulsion, characterized by the fact that water is added once, above the phase inversion point, to a mixture of polyorganosiloxane and surfactant, this is mixed together and stirred, then this is diluted by adding the remaining water.

2. A silicone emulsion obtained by the manufacturing method according to Claim 1, wherein the emulsion average particle diameter is 1-100 µm.

3. A hair cosmetic material characterized by the fact that it contained the silicone emulsion according to Claim 2.

Detailed explanation of the invention

[0001]

Technical field of the invention

The present invention pertains to an O/W silicone emulsion to be used in various fields for the purposes of producing luster, antifoaming, mold parting, lubrication and tactile improvement, for example, and a method for its manufacture. More specifically, it pertains to a silicone emulsion suitable for use in the fields of cosmetics and perfumery for the purpose of tactile improvement, for example, and a method for its manufacture.

[0002]

Prior art and problems to be solved by the present invention

Conventionally, silicone emulsions have often been used for the purpose of giving sensations, such as luster and smoothness, for example, to hair and skin. When the characteristics of silicone are conferred,

* [Numbers in right margin indicate pagination of the original text.]

there are advantages such as rapid adhesion to hair and skin, and the advantage of not dripping during use, and the composition raises the anticipation that excellent properties will be obtained in proportion to the size of the emulsion particle diameter, but when the particle diameter increases, the emulsion stability becomes degraded, and, when blended into various commercial articles, there is the problem of their blending stability being significantly degraded. Emulsions generally have a tendency for their stability to become poor in proportion to the particle diameter size, which is thought to be due to the fact that any attempt to increase the particle diameter results in a wider particle diameter distribution, often resulting in particle diameter distributions with multiple peaks, which causes an extreme lowering of stability. Therefore, that which has the relatively small particle diameter of less than 1 μm as the particle diameter of the silicone emulsion is presently used. Until now, several methods are known for the purpose of controlling the emulsion particle diameter. For example, there is the method of selecting the HLB or type of surfactant. In this case, as seen from the multitude of types of surfactants presently sold commercially, there is a problem in that there is an extremely large number of points to be investigated in order to obtain an emulsion with the target particle diameter together with the silicone to be used, and, therefore, a long period of time is required for investigation. Conversely, in the fields of cosmetics/perfumes, the surfactants that can be used is limited, so it is difficult to obtain an emulsion with satisfactory properties just by combining these. Various methods have been proposed as specific methods for controlling emulsion particle diameter, such as the HLB method (Griffin, W.C.; *J. Soc. Cosmet. Chem.*, 1:311, 1949), liquid crystal emulsion method (Washiya et al., *Yukagaku [Journal of the Japan Oil Chemists' Society]*, 30, 38, 1981), phase inversion emulsification (T. J. Lin, *J. Soc. Cosmet. Chem.*, 30, 167, [1]976), and D-phase emulsification (Washiya et al., *Nippon Kagaku Kaishi [Journal of the Chemical Society of Japan]*, 1399, 1983). However these emulsification methods are mainly for obtaining articles of small particle diameters of less than 1 μm, and cannot be effective means for

controlling the particle diameter of articles with large particle diameter as in the present invention. Obviously, there are cases where things greater than 1 μm can be obtained, depending on the surfactant used, but there was the problem with stability described above. It is possible to think of other methods for controlling particle diameter by changing the share pertaining to the emulsification time, such as changing the temperature during emulsification, changing the type of emulsification device, or changing the agitation rate during emulsification. However, with these methods, there is the difficulty of accurately controlling the temperature or agitation rate and also the share pertaining to the emulsification time is by no means homogeneous throughout the entire emulsification system, thus partial differences appear such as the agitation vane circumference, the broad distribution of particle diameters in the resulting emulsion, and a particle diameter distribution with multiple peaks. When this happens, the emulsion and blending stability with other materials is significantly degraded. This tendency is particularly pronounced for larger particle diameter articles. This is because the emulsion particle diameter becomes larger in inverse proportion to the share pertaining to the emulsification time, but it is difficult to make a small share completely homogeneous. Furthermore, as the scale of manufacturing increases, it becomes more difficult to bestow a homogeneous share throughout the entire emulsification system, so when manufacturing equipment or the scale is altered, even if conditions such as temperature or [agitator] revolution rate are identical, there are many instances of differing particle diameters, thus a necessity emerges to investigate the manufacturing conditions for each occasion, such as optimal temperature or revolution rate.

[0003]

Object of the invention

The object of the invention is to obtain an emulsion that has a narrow distribution of obtained particle diameters that has excellent emulsion stability and blending stability while also having large particle diameter, which can be easily controlled without changing the emulsion composition, which solves the aforementioned problems.

[0004]

Structure of the invention

As a result of conducting a series of diligent investigations to accomplish the object described above, the inventors discovered that it was possible to obtain an emulsion with a narrow distribution of particle diameters by adding water exceeding the phase inversion point to a mixture of silicone and surfactant, then, mixing and stirring, then diluting with the remaining water, thus arriving at the present invention. More specifically, the present invention is a method for manufacturing a polyorganosiloxane O/W silicone emulsion in which water is added, above the phase inversion point to a mixture of polyorganosiloxane and surfactant, mixed together and stirred, then diluted by adding the remaining water; a silicone emulsion obtained by said manufacturing method, wherein the emulsion average particle diameter is 1-100 μm; and a hair cosmetic material that contains said silicone emulsion. /3

[0005]

Working mode of the invention

The following statements explain the invention in greater detail. Any publically known polyorganosiloxane can be used in the present invention. That described by the general formula:

$$R_aSiO_{(4-a)/2}$$

(where R is a substituted or unsubstituted monovalent hydrocarbon group, a is a positive number less than 4) may be mentioned as an example. In the formula, R is a substituted or unsubstituted monovalent hydrocarbon group, which may be the same or many different types; examples include alkyl groups such as methyl, ethyl, hexyl, and decyl; aryl groups such as phenyl; aralkyl group such as 2-methyl phenylethyl; amino-containing groups such as 3-aminopropyl and 2-aminoethyl-3-aminopropyl; polyoxyalkylene groups such as polyoxyethylene-polyoxypropylene; and fluorine-containing groups such as 3,3,3-trifluoropropyl. These may be selected accordingly to fit the application; for example, in the case of hair cosmetics, a selection may be made amongst various R groups as follows: a methyl group is desirable when the purpose is to impart a smooth feeling to the hair; an amino-containing group is desirable for the purpose of improving adherence and a moist feeling conferring effect; and an aryl group is desirable for the purse of improving luster. Also, a is a positive number less than 4, preferably 1.0-2.5 in terms of the ease of emulsification and utilization of the obtained siloxane. The polyorganosiloxane used by be a single type or a multiplicity of types. For example, assuming a polydimethyl siloxane that has a viscosity exceeding 1,000,000 cSt, the effect of cuticle protection and an excellent tactile sensation to the hair, diffuses poorly into the hair when used alone, the general practice is to supplement this with, for example, several hundred cP polydimethyl siloxane. The use of, an isoparaffin-type solvent also exists, instead of a polydimethyl siloxane of several hundred cSt. It is also possible to suitably use the invented manufacturing method in this case. The use of a polydimethyl siloxane having a viscosity of 10,000-100,000,000 cST is preferred, and a viscosity of 50,000-50,000,000 is particularly preferred, in terms of making it possible to efficiently obtain an emulsion with good stability and small distribution of particle diameters when using the invented

manufacturing method, and when the obtained emulsion composition is used in a hair cosmetic, to obtain a good cuticle protection effect and a good tactile sensation to the hair.

[0006]

The surfactant used in the present invention may be a nonionic, anionic or a cationic surfactant. Specific examples of nonionic surfactants include glyceryl fatty acid esters such as glyceryl monolaurate, glyceryl monomyristate, glyceryl monostearate, and glyceryl monooleate; that having a similar fatty acid residue, such as glycerine fatty acid ester, sorbitan fatty acid ester, polyoxyethylene glycerine fatty acid ester, polyoxyethylene sorbitan fatty acid ester, and polyoxyethylene fatty acid ester; polyoxyethylene alkyl ethers such as polyoxyethylene lauryl ether, polyoxyethylene cetyl ether, polyoxyethylene stearyl ether, and polyoxyethylene oleyl ether; polyoxyethylene alkylphenyl ethers such as polyoxyethylene octylphenyl ether and polyoxyethylene nonylphenyl ether; oxyethylene/oxypropylene copolymer; and polyether-modified polydimethyl siloxane. Specific examples of anionic surfactants include sodium alkylbenzene sulfonates such as sodium hexylbenzene sulfonate, sodium octylbenzene sulfonate, sodium decylbenzene sulfonate, sodium dodecylbenzene sulfonate, sodium cetylbenzene sulfonate, and sodium myristylbenzene sulfonate; sodium alkylnaphthyl sulfonates such as sodium butylnaphthyl sulfonate; polyoxyethylene alkyl ether sulfate ester sodium salts such as sodium polyoxyethylene octyl ether sulfate ester, sodium polyoxyethylene decyl ether sulfate ester, and sodium polyoxyethylene eicosyl ether sulfate ester; and polyoxyethylene mono (alkylphenyl) ether sulfate ester sodium salts such as sodium polyoxyethylene decylphenyl ether sulfate ester. Specific examples of cationic surfactants include quaternary ammonium salts such as octyl trimethyl ammonium chloride, dodecyl trimethyl ammonium chloride, hexadecyl trimethyl ammonium chloride, beef tallow trimethyl ammonium chloride, palm oil trimethyl ammonium chloride, octyl dimethylbenzyl ammonium chloride, decyl

dimethylbenzyl ammonium chloride, and dioctadecyldimethyl ammonium chloride. These surfactants can ordinarily be used in combinations of 2 or more types within the same or different system. In the invented manufacturing method, the use of an item that contains at least 1 or more types of nonionic surfactant is preferred, due to the impartation of favorable sensation to the hair when an emulsion with favorable stability and narrow particle diameter distribution is obtained, or when the obtained emulsion composition is used in a hair cosmetic, and the use of only nonionic surfactants is particularly preferable. Of the previously mentioned nonionic surfactants, the use of polyoxyethylene sorbitan fatty acid ester, polyoxyethylene fatty acid ester, and polyoxyethylene alkyl ether is preferred. The surfactant is to be used in the amount of 1-30 parts by weight per 100 parts by weight of the polyorganosiloxane. This is because emulsification will be inadequate when less than 1 part by weight is used, and because the intrinsic properties of silicone will not be sufficiently expressed due to the large amount of surfactant if an emulsion is used where [the amount of surfactant] exceeds 30 parts by weight. It is preferred that [the amount of surfactant] be 2-20 parts by weight, more preferably 5-15 parts by weight, per 100 parts by /4
weight of polyorganosiloxane.

[0007]

Conventionally, and ordinarily, when an O/W emulsion is prepared, the oil phase component and surfactant are heated as required, mixed together, and further mixed while water is gradually added, such that [the emulsion] is obtained. In this case, a colloid mill, homomixer, pressurized homogenizer, or other emulsification machine is used. For example, in the phase inversion emulsification method described as the prior art, water is gradually added to the mixture of oil phase component and surfactant. First, having passed through a W/O type, the formation of a D-phase is brought about. The D-phase, which is also called the surfactant phase, is a liquid crystal phase in which the oil and water are arranged

in sandwich form via a surfactant lamellar phase, thus presenting a transparent appearance. At this time, the continuous phase not influenced by the oil phase, which is the dispersion phase becomes the D-phase. Water is further added, making an O/W emulsion. When this is done, an O/W emulsion in which the oil droplets are finely dispersed is obtained. In this method, water is gradually added, and homogeneity is obtained within the emulsification system at each stage, such that an attempt is made to obtain an O/W emulsion with homogeneously small particle diameters; more specifically, the number of times that water is charged is 3 or more times, ordinarily 5-10 times. Thorough stirring is required in order to obtain homogeneity within the system at each occasion, so a long period of time is required for manufacture. Ordinarily the D-phase is a hard, gelatinous substance, which requires high agitation efficiency and enormous motive force in order to be homogenized, so as the material to be emulsified becomes more viscous, the homogenization of the system becomes more problematic. The presence of partially stirred insufficient regions occurs, resulting in varied particle diameter. When the equipment or scale are changed, the particle diameter distribution of the emulsion varies, which is difficult to control. The amount of water at the phase inversion point can be measured by various methods, such as repeating a process of adding small amounts of water to previously mixed oil phase component and surfactant while maintaining a constant temperature, and then, assessing the point of maximum transparency within the contents as the phase inversion point.

[0008]

The present invention is characterized by the fact that water in excess of the phase inversion point is added once to the mixed system of oil phase component and surfactant, and this water charge volume is in excess of the amount that results in the aforementioned D-phase. Depending on the oil phase component and surfactant that are used, the quantity of water causing the formation of a D-phase will

differ, so it is difficult to [define] limits, such as to regulate the water charge volume with respect to the oil phase component. In the present invention, the amount of water at the phase inversion point is taken as the standard, and 1-5 times this amount is the initial charge of water. If the initial charge of water is less than 1 times this amount, even if is added as diluent water, the homogeneous dispersion of the mixture of water with the oil phase component and surfactant will become impossible, or a long period of time will be required for dispersion. If the initial charge of water is more than 5 times this amount, as expected, either it will be impossible for the mixture of oil phase component and surfactant to be dispersed into the water, or a long period of time will be required for dispersion. In order to obtain a homogeneous distribution of particle diameters, and for easy dispersion in both the water charging process and the dilution process, it is preferred that the amount of water be 1.1-3 times, more preferably 1.5-2 times. According to the present invention, the diameter of the obtained particles will be small when the charging of water in the phase inversion point water volume is followed by diluent water, and the diameter of the obtained particles will gradually become larger as the volume of water for the first water charge is increased above the phase inversion point. In other words, it becomes possible to control the obtained emulsion particle diameter by varying the volume of water for the initial charge. The present invention makes it possible to shorten the manufacturing process because the water charging is divided into just 2 charges. Furthermore, homogeneous mixing within the system is easy because there is no formation of a hard gelatinous phase. In other words, [the invented method] is not easily susceptible to the influence of equipment and scale. [In conclusion,] this method has a small number of manufacturing processes, and it is not susceptible to the influence of equipment and scale, so it is a method that is suitable for either batch manufacturing or continuous manufacturing.

[0009]

In the invented method for manufacturing the emulsion, it is possible to conduct emulsification conjointly using polyorganosiloxane [and] an isoparaffinic solvent, as described above, and the following mentioned materials may be blended in such a conjoint utilization to impart a wetness widening characteristic, diluting the polyorganosiloxane, when the obtained emulsion is used, for example, in a hair cosmetic: paraffinic solvents such as hexane and heptane, aromatic solvents such as toluene and xylene, isoparaffinic solvents such as isoheptane and isooctane, chlorine solvents such as chloroform, as well as ester type solvents and glycol type solvents. The combined use of silica powder is also possible for the purpose of anti-foaming of the obtained emulsion, or zeolite and other solid powders for thixo-properties, waxes such as carnauba wax to improve luster; furthermore it is possible to use preservatives such as propyl p-hydroxybenzoate, sodium benzoate, citric acid and salicylic acid, or viscosity increasing agents such as alginic acid, gum arabic, cassein, methyl cellulose, hydroxyethyl cellulose, cation modified etherified cellulose, hydroxypropyl starch, polyvinyl alcohol, polyvinyl pyrrolidone, and bentonite. The invented manufacturing method can apply even if these substances are used together with polyorganosiloxane.

[0010]

The emulsion obtained by the invented manufacturing method has the characteristic that the particle diameter distribution is monodisperse and narrow, and the characteristic that the stability of the emulsion is excellent. Therefore, when it is used as a material for cosmetics and the like, it has excellent maintenance characteristics of the obtained cosmetic article. With the invented manufacturing method, it is possible to efficiently obtain an emulsion for which the average particle diameter is generally 0.1-200 μm, and it is possible to obtain, with good efficiency, relatively large particle diameters, for

example of 1-100 μm, and 3-80 μm in particular, which, until now, have been difficult to obtain in a manner with good stability. Such an emulsion with large particle diameter that has been obtained by the invented manufacturing method not only has excellent stability, but also has the characteristic of exhibiting the excellent properties intrinsic to silicone when it is blended into cosmetics and similar articles. /5

[0011]

Effect of the invention

The invented manufacturing method makes it possible to obtain a silicone emulsion with good stability and a narrow particle diameter distribution and has the particular characteristic that a silicone emulsion is obtained that has good stability with large particle diameter which has heretofore been problematic to manufacture. It is further characterized by the ease with which the particle diameter of the emulsion is controlled, the low susceptibility to the type of equipment or scale, and a manufacturing process that is shorter than the conventional manufacturing method.

[0012]

Application examples

The following statements describe the present invention in greater detail. In these application examples, "%" indicates "weight %." The value for viscosity is 25°C. The emulsion compositions used in the application examples are shown in Table 1. The particle diameter of the emulsions was measured by the method indicated below. The phase inversion point at 70°C was measured by the method indicated below. The phase inversion point water quantity obtained here is shown in Table 1.

[0013]

Table 1

	①	②	③	④	⑤
	成分	組成－1	組成－2	組成－3	組成－4
⑥	ポリジメチルシロキサン1	36%	36%	44%	—
	ポリジメチルシロキサン2	—	—	—	60%
	ポリジメチルシロキサン3	24%	24%	30%	—
	アミノ基含有シロキサン	—	6%	—	—
⑦	ノニオン系界面活性剤1	1.2%	1.8%	—	1.5%
	ノニオン系界面活性剤2	—	1.1%	1.5%	—
	ノニオン系界面活性剤3	3.8%	3.1%	—	2.0%
	ノニオン系界面活性剤4	—	—	—	2.5%
	カチオン系界面活性剤1	—	—	0.5%	—
⑧	水	35%	28%	24%	34%
⑨	転相点水量	7%	7%	5%	9%

Key: 1 Ingredient

2 Composition 1

3 Composition 2

4 Composition 3

5 Composition 4

6 Polydimethyl siloxane 1

Polydimethyl siloxane 2

Polydimethyl siloxane 3

Siloxane containing amino groups

7 Nonionic surfactant 1

Nonionic surfactant 2

Nonionic surfactant 3

Nonionic surfactant 4

Cationic surfactant 1

8 Water

9 Phase inversion point water quantity

[0014]

Notes)

Polydimethyl siloxane 1: 200 cSt polydimethyl siloxane

Polydimethyl siloxane 2: 100,000 cSt polydimethyl siloxane

Polydimethyl siloxane 3: 2,000,000 cSt polydimethyl siloxane

Siloxane containing amino groups: 1000 cSt, polydimethyl siloxane that contains 2-aminoethyl-3-aminopropyl groups with a nitrogen atom content of 0.9%

Nonionic surfactant 1: polyoxyethylene (23) lauryl ether

Nonionic surfactant 2: polyoxyethylene (6) lauryl ether

Nonionic surfactant 3: polyoxyethylene (55) monostearate

Nonionic surfactant 4: polyoxyethylene (20) sorbitan monostearate

Cationic surfactant 1: cetyl trimethyl ammonium chloride

-- Emulsion particle diameter measurement

A Coulter-manufactured, LS-model (measurement principle: laser diffraction/scattering technology) was used. The average particle diameter, modal particle diameter, and average particle diameter/modal

particle diameter were confirmed from the obtained results. The particle diameter distribution spread was determined from the average particle diameter/modal particle diameter ratio. In other words, as the ratio approached 1, the distribution was monodisperse and narrow.

-- Measurement of the phase inversion point water quantity

This siloxane ingredient and active ingredient were heated to 70°C and stirred, water was repeatedly added in increments of 1% volume and stirred. The point at which the contents were the most transparent was visually determined, and the amount of water added [up until] that time was taken as the phase inversion point water quantity.

[0015]

Application Example 1

The various ingredients were used such that the total of emulsion composition 1 was 100 g. A mixture of 36 g of polydimethyl siloxane 1 and 24 g of polydimethyl siloxane 3 was placed in a 200 cc flask and heated to 70°C. This 70°C heating continued until all water was added. Here, 1.2 g of nonionic surfactant 1 and 3.8 g of nonionic surfactant 3 were added, and this was manually stirred using a metal spatula. While maintaining the 70°C heating, stirring was continued for approximately 5 min, when the /6 nonionic surfactant melted and was mixed with the polydimethyl siloxane. Here, 8 g of water was added as the initial charge of water. Stirring was continued for approximately 10 min, and a transparent gelatinous substance was obtained. Then, the remaining 27 g of water was added as the diluent water, the heating to 70°C was stopped, and as [the liquid] was allowed to cool, it was stirred for approximately 10 min, giving a milky white emulsion. The average particle diameter, modal particle diameter, and average particle diameter/modal particle diameter of the emulsion is shown in Table 2.

Application Example 2

An operation identical to that of Application Example 1 was conducted, with the exception that the initial charge of water was 10 g and the amount of diluent water was 25 g. After addition and stirring of the initial charge of water, a viscous, slightly transparent, milky white liquid, and finally a milky white emulsion, was obtained. The particle diameter and other evaluation results are shown in Table 2.

Application Example 3

An operation identical to that of Application Example 1 was conducted, with the exception that the initial charge of water was 12 g and the amount of diluent water was 23 g. After addition and stirring of the initial charge of water, a viscous, slightly transparent, milky white liquid, and finally a milky white emulsion, was obtained. The particle diameter and other evaluation results are shown in Table 2.

Application Example 4

An operation identical to that of Application Example 1 was conducted, with the exception that the initial charge of water was 15 g and the amount of diluent water was 20 g. After addition and stirring of the initial charge of water, a viscous, milky white liquid, and finally a milky white emulsion, was obtained. The particle diameter and other evaluation results are shown in Table 2.

Application Example 5

An operation identical to that of Application Example 1 was conducted, with the exception that the initial charge of water was 18 g and the amount of diluent water was 17 g. After addition and stirring of the initial charge of water, a viscous, milky white liquid, and finally a milky white emulsion, was obtained. The particle diameter and other evaluation results are shown in Table 2.

Application Example 6

An operation identical to that of Application Example 1 was conducted, with the exception that the initial charge of water was 22 g and the amount of diluent water was 13 g. After addition and stirring of the initial charge of water, a viscous, milky white liquid, and finally a milky white emulsion, was obtained. The particle diameter and other evaluation results are shown in Table 2.

Application Example 7

The various ingredients were used such that the total [amount] of emulsion composition 1 was 1500 g. A mixture of 540 g of polydimethyl siloxane 1 and 360 g of polydimethyl siloxane 3 was placed in an "Ajit-Homomixer" (Tokushu Kika Kogyo Co., Ltd.) equipped with a 2-liter vessel and heated to 70°C. Heating was continued until the diluent water had been added. Also, mixing was continued with a 60 rpm rate for the paddle mixer. Mixing conditions were continued until the final emulsion was taken out. Into this was added 18 g of nonionic surfactant 1 and 48 g of nonionic surfactant 3 and stirred continuously for approximately 20 min; the nonionic surfactant had melted and was mixed with the polydimethyl siloxane. Here, 225 g of water was added as the initial charge of water, stirring was continued for approximately 20 min and a transparent gelatinous substance was obtained. Then, the remaining 300 g of diluent water was added and stirred for approximately 20 min, thus preparing a milky white emulsion, which was evaluated in the same was as that of Application Example 1. Application Example 7 changes the apparatus in Application Example 4 and increase the scale.

Application Example 8

A milky white emulsion was obtained by an operation identical to that of Application Example 7, with the exception that the revolution rate of the paddle mixer was 15 rpm. This was evaluated in identical fashion.

[0016]

Comparative Example 1

The various ingredients were used such that the total of emulsion composition 1 was 100 g. A mixture of 36 g of polydimethyl siloxane 1 and 24 g of polydimethyl siloxane 3 was placed in a 200 cc flask and heated to 70°C. This 70°C heating was continued until the entire amount of water had been added. Here, 1.2 g of nonionic surfactant 1 and 3.8 g of nonionic surfactant 3 were added and manually stirred using a metal spatula. While maintaining the 70°C heating, stirring was continued for approximately 5 min, when the nonionic surfactant melted and was mixed with the polydimethyl siloxane. Here, 2 g of water was added as the initial charge of water, and stirring was continued for approximately 10 min. Then, the operation of adding 2 g of water and stirring for 10 min was repeated 2x. Up until this point, the substance had a viscous, milky white oily form, but here it became a transparent gelatinous substance. The operation of adding 2 g of water and stirring for 10 min was repeated 8x. Then, 13 g of water was added, the heating to 70°C was stopped, and as [the substance] was allowed to cool, stirring continued, thus a milky white emulsion was prepared. This was evaluated in a manner identical to Application Example 1.

Comparative Example 2

An emulsion was obtained by an operation identical to that of Comparative Example 1, with the exception that heating to 70°C was stopped after the stirring that following the initial charge of water. This was evaluated in identical fashion.

Comparative Example 3

The various ingredients were used such that the total of emulsion composition 1 was 1500 g. A mixture of 540 g of polydimethyl siloxane 1 and 360 g of polydimethyl siloxane 3 was placed in an "Ajit-Homomixer" (Tokushu Kika Kogyo Co., Ltd.) equipped with a 2-liter vessel and heated to 70°C. This heating was continued until the diluent water had been added. Also, mixing was continued with a 60 rpm rate for the paddle mixer. These mixing conditions were continued until the final emulsion was taken out. 18 g of nonionic surfactant 1 and 48 g of nonionic surfactant 3 was added and stirred continuously for approximately 20 min; the nonionic surfactant had melted and was mixed with the polydimethyl siloxane. Here, 30 g of water was added as the initial charge of water, and stirring was continued for approximately 20 min. The operation of adding 30 g of water and stirring for 20 min was repeated 2x. Up until this point, the substance had a viscous, milky white oily form, but here it became a transparent gelatinous substance. The operation of adding 30 g of water and stirring for 20 min was repeated 8x. Then, 195 g of diluent water was added, the heating to 70°C was stopped, and stirring continued as [the substance] was allowed to cool, thus a milky white emulsion was prepared. This was evaluated in a manner identical to Application Example 1. Comparative Example 3 changes the apparatus in Comparative Example 1 and increases the scale. /7

Comparative Example 4

An emulsion was obtained by an operation identical to that of Comparative Example 3, with the exception that the revolution rate of the paddle mixer was 15 rpm. This was evaluated in identical fashion.

Comparative Example 5

An operation identical to that of Application Example 1 was conducted, with the exception that the initial charge of water was 4 g and the amount of diluent water was 31 g. After addition and stirring of the initial charge of water, a viscous, slightly transparent, milky white liquid was obtained, but after the addition and stirring of diluent water, the diluent water did not blend in, thus no milky white emulsion could be obtained.

[0017]

Application Example 9

The various ingredients were used such that the total of emulsion composition 2 was 100 g. A mixture of 36 g of polydimethyl siloxane 1, 24 g of polydimethyl siloxane 3, and 6 g of amino group-containing siloxane was placed in a 200 cc flask and heated to 70°C. Here, 1.8 g of nonionic surfactant 1, 1.1 g of nonionic surfactant 2, and 3.1 g of nonionic surfactant 3 were added, and this was manually stirred using a metal spatula. Stirring was continued for approximately 5 min, when the nonionic surfactant melted and was mixed with the polydimethyl siloxane. Here, 8 g of water was added as the initial charge of water, and stirring was continued. When stirring had continued for approximately 10 min, [the substance] became a transparent gelatinous substance. Then, the remaining 20 g of diluent water was added and stirred for approximately 10 min, thus giving a milky white emulsion. This was evaluated in the same manner as Application Example 1.

Application Example 10

An operation identical to that of Application Example 9 was conducted, with the exception that the initial charge of water was 20 g, and the amount of diluent water was 8 g. After addition and stirring of the initial charge of water, a viscous, milky white liquid, and finally a milky white emulsion, was obtained. The particle diameter and other evaluation results are shown in Table 2.

Application Example 11

The various ingredients were used such that the total of emulsion composition 3 was 100 g. A mixture of 44 g of polydimethyl siloxane 1 and 30 g of polydimethyl siloxane 3 was placed in a 200 cc flask, to which 1.5 g of nonionic surfactant 2 and 0.5 g of cationic surfactant 2 were added, and this was manually stirred using a metal spatula. Stirring was continued for approximately 5 min. Here, 6 g of water was added as the initial charge of water, and stirring was continued. When stirring had continued for approximately 10 min, [the substance] became a transparent gelatinous substance. Then, the remaining 18 g of diluent water was added, and this was stirred for approximately 10 min, thus giving a milky white emulsion. This was evaluated in the same manner as Application Example 1.

Application Example 12

An operation identical to that of Application Example 11 was conducted, with the exception that the initial charge of water was 15 g and the amount of diluent water was 9 g. After addition and stirring of the initial charge of water, a viscous, milky white liquid, and finally a milky white emulsion was obtained. The particle diameter and other evaluation results are shown in Table 2.

Application Example 13

The various ingredients were used such that the total of emulsion composition 4 was 100 g. 60 g of polydimethyl siloxane 2 was placed in a 200 cc flask and heated to 70°C. Here, 1.5 g of nonionic surfactant 1, 2 g of nonionic surfactant 3, and 2.5 g of nonionic surfactant 4 were added, and this was manually stirred using a metal spatula. Stirring was continued for approximately 5 min. Here, 10 g of water was added as the initial charge of water, and stirring was continued. When stirring had continued for approximately 10 min, [the substance] became a transparent gelatinous substance. Then, the remaining 24 g of diluent water was added, and stirred for approximately 10 min, thus giving a milky white emulsion. This was evaluated in the same manner as Application Example 1.

Application Example 14

An operation identical to that of Application Example 13 was conducted, with the exception that the initial charge of water was 15 g and the amount of diluent water was 19 g. After addition and stirring of the initial charge of water, a viscous, milky white liquid, and finally a milky white emulsion, was obtained. The particle diameter and other evaluation results are shown in Table 2.

[0018]

Table 2

/8

	No	① 平均粒子径 (μm)	② 最頻粒子径 (μm)	③ 平均粒子径／最頻粒子径
④	実施例1	1.64	1.59	1.03
	実施例2	2.56	2.54	1.01
	実施例3	3.25	3.14	1.04
	実施例4	3.75	3.69	1.02
	実施例5	5.99	6.45	0.92
	実施例6	6.43	7.08	0.91
	実施例7	3.65	3.45	1.06
	実施例8	3.82	3.70	1.03
⑤	比較例1	5.51	4.71	1.17
	比較例2	1.60	1.51	1.06
	比較例3	3.64	3.57	1.02
	比較例4	7.81	16.1	0.62
⑥	実施例9	1.78	1.81	0.98
	実施例10	7.34	7.59	0.97
	実施例11	24.5	22.5	1.09
	実施例12	46.4	42.8	1.08
	実施例13	11.4	10.3	1.11
	実施例14	56.8	54.9	1.03

Key: 1 Average particle diameter (μm)

2 Modal particle diameter (μm)

3 Average particle diameter/modal particle diameter

4 Application Example 1

Application Example 2

Application Example 3

Application Example 4

Application Example 5

Application Example 6

Application Example 7

Application Example 8

5 Comparative Example 1

Comparative Example 2

Comparative Example 3

Comparative Example 4

6 Application Example 9

Application Example 10

Application Example 11

Application Example 12

Application Example 13

Application Example 14

[0019]

In Application Examples 1-6 the quantity of the initial charge of water was varied, the resulting particle diameter changed and the distribution of this was small. The blend used in Application Example 7 was the same as in Application Example 4, but the equipment and scale changed, obtained particle diameter distribution was the same. The same equipment and scale were used in Application Example 8 as in Application Example 7, but the revolution rate of the agitator was varied, yet again the obtained

particle diameter distribution was the same. In contrast, even though the particle diameter obtained by emulsification temperature varied in Comparative Examples 1 and 2, the distribution was wide in Comparative Example 1, which had a high temperature. Comparative Example 3 used the same blend and temperature as Comparative Example 1, varied the equipment and scale, but obtained a small particle diameter. In Comparative Example 4, which used the same equipment and scale as Comparative Example 3 but varied the revolution rate of the agitator, the obtained particle diameter was large, and its distribution was extremely wide. In Application Examples 9-14, it was possible to vary the particle diameter by varying the amount of initial water charge, even with different systems of surfactant and polyorganosiloxane, and the particle diameter distribution was relatively narrow.

[0020]

The following statements describe application examples in which the invented emulsion is used in cosmetics.

Application Example 15

Hair setting composition

The emulsion prepared in Application Example 1 was used to prepare a hair setting composition having the composition shown below. This was evaluated, and the results are shown in Table 3.

-- Composition

Silicon emulsion (Application Example 1)	5.0%
Hydroxyethyl cellulose	0.2%
Ethyl alcohol	10.0%

Fragrances	0.2%
Ion exchange water	84.6%

-- Evaluation method

Suppleness: 4 g of hair setting composition was evenly coated from a distance of 20 cm onto 2 g of hair 25 cm long. This was then neatly combed with a commercially available comb before drying. The hair was then, dried for 60 min by warm air at 40°C and evaluated according to the following scoring criteria.

O No rigidity when touched by the hand; [rather,] a natural tactile sensation.

Δ Somewhat rigid feeling when touched by the hand.

X Extremely rigid feeling when touched by the hand, a hard tactile sensation.

Smoothness: 4 g of hair setting composition was evenly coated from a distance of 20 cm onto 2 g of hair 25 cm long. The hair was then, dried for 60 min by warm air at 40°C, after which the comb-passage condition, using a commercially available comb, was evaluated according to the following scoring criteria.

O No snagging, comb passes smoothly through

Δ Comb passes through, but with snagging.

X Comb cannot pass through.

Set holdability: 4 g of hair setting composition was evenly coated from a distance of 20 cm onto 2 g /9
of hair 25 cm long. This hair was then wrapped around a curler of 1.2 cm outer diameter, and dried for 60 min by warm air at 40°C. The hair was then removed from the curler and draped perpendicularly in an atmosphere of 80% relative humidity and 30°C temperature, the length was measured immediately (L1), and after 1 h of standing, (L2). The curl retention was then calculated by the following formula.

curl retention (%) = (25-L2) / (25 - L1) x 100

A curl retention of 60% or greater was to be acceptable (indicated by "O" in the table), and less than 60% to be unacceptable (indicated by "X" in the table).

[0021]

Application Example 16

A hair setting composition was prepared identical to Application Example 15, with the exception that the emulsion prepared in Application Example 4 was used. This was evaluated. The results are shown in Table 3.

Application Example 17

A hair setting composition was prepared identical to Application Example 15, with the exception that the emulsion prepared in Application Example 7 was used. This was evaluated. The results are shown in Table 3.

Application Example 18

A hair setting composition was prepared identical to Application Example 15, with the exception that the emulsion prepared in Application Example 9 was used. This was evaluated. The results are shown in Table 3.

Application Example 19

A hair setting composition was prepared identical to Application Example 15, with the exception that the emulsion prepared in Application Example 11 was used. This was evaluated. The results are shown in Table 3.

Comparative Example 6

A hair setting composition was prepared identical to Application Example 15, with the exception that the emulsion prepared in Comparative Example 1 was used. This was evaluated. The results are shown in Table 3.

Comparative Example 7

A hair setting composition was prepared identical to Application Example 15, with the exception that the emulsion prepared in Comparative Example 4 was used. This was evaluated. The results are shown in Table 3.

[0022]

Table 3

		1 実施例					2 比較例	
		15	16	17	18	19	6	7
3	しなやかさ	○	○	○	○	○	×	○
	なめらかさ	○	○	○	○	○	△	△
	セット保持力	○	○	○	○	○	○	×

Key: 1 Application Examples

2 Comparative Examples

3 Suppleness

Smoothness

Set holdability

[0023]

Application Example 20 and Comparative Example 8 (Shampoo Composition)

2 types of shampoo compositions having the composition shown below were prepared, using the emulsion prepared in Application Example 1 or that of Comparative Example 1. These were then evaluated.

-- Composition

Silicone emulsion (Application Example 1 or Comparative Example 1)	3.0%
Polyoxyethylene lauryl ether	15.0%
Lauric acid diethanol amide	5.0%
Fragrances	0.2%
Ion exchange water	76.8%

The shampoo composition using the emulsion of Application Example 1 (i.e., Application Example 20) gives a nice luster to the hair, is able to confer a refreshing feeling, and has favorable stability. In contrast, the shampoo composition using the emulsion of Comparative Example 1 (i.e., Comparative Example 8) is inadequate in terms of giving luster or stability to the hair, although it is able to give a somewhat good degree of sensation.